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(54) **FINAL-DRIVE LUBRICATING OIL COMPOSITION.**

(57) A final-drive lubricating oil composition comprising a base oil and, added thereto, (A) 0.1-5 wt.% of a phosphoric ester, a phosphorous ester or an alkylamine salt of a phosphoric ester, (B) 0.1-5 wt.% of an organic acid represented by the general formula:  $R^1\text{-CON}(\text{CH}_3)\text{CH}_2\text{COOH}$  (wherein  $R^1$  represents  $\text{C}_1\text{-C}_{18}$  alkyl or  $\text{C}_2\text{-C}_{18}$  alkenyl) and (C) 3-15 wt.% of a sulfurized olefin or an alkyl sulfide, each based on the whole composition. The composition serves to further improve the transmission efficiency at the differential gear under varied conditions of input torque, input rotational speed and oil temperature as compared with the conventional compositions containing additives based on sulfur and phosphorous compounds, thus leading to fuel consumption improvement.

**EP 0 677 570 A1**

## Field of the Invention

The present invention relates to a novel lubricating oil composition for use in a final drive, and more particularly to a lubricating oil composition for use in a final drive using in a car of a front-engine and rear-wheel-drive system or the like, which composition improves the efficiency of transmission in a differential gear under various conditions such as the input torque, the input number of revolutions and the temperature of the oil, as compared with the conventional ones comprising S-containing and P-containing additives blended therein.

## Prior Art

The final drive of a car has a function (1) of further decreasing a driving force decreased by a transmission and rectangularly changing the direction of transmission of the force, and a differential function (2) of securing smooth driving even when a difference in revolution arises between right and left driving wheels at the time of turning the car around. The former function is performed by a decelerator, while the latter function is performed by a differential gear. The decelerator is constituted of a decelerating pinion and a decelerating gear wheel. Hypoid gears are generally used as such decelerating pinion and gear wheel.

On the other hand, the differential gear (differential) is constituted of two or four differential pinions and two differential gear wheels engaged therewith, which are built in a differential gear box. The differential pinions, which are connected to the differential gear box by means of a shaft, is capable of orbital revolution together - with the gear box while revolving on its own axis around the shaft. Consequently, a difference in revolution between the interior and exterior driving wheels can be mechanistically absorbed even if such a difference arises when the car is turned around.

The hypoid gears of a gear transmission mechanism as is used in the foregoing final drive of the car are exposed to severe conditions such as a high speed of revolution and a heavy load, leading to the necessity for a gear oil excellent in seizing-proofing and abrasion-proofing properties. Accordingly, use is generally made of a gear oil comprising a base oil admixed with a sulfur-containing extreme-pressure additive such as an olefin sulfide or an alkyl sulfide and a phosphorus-containing extreme-pressure additive such as a phosphoric ester, a phosphorous ester or an alkylamine salt of a phosphoric ester, wherein the sulfur-containing extreme-pressure additive improves the above-mentioned seizing-proofing properties while the phosphorus-containing extreme-pressure additive improves the above-mentioned abrasion-proofing properties.

In the case of such a conventional gear oil comprising S-containing and P-containing additives blended therein to impart thereto extreme-pressure properties and abrasion-proofing properties, however, the efficiency of transmission in a differential gear is about 90 to 95% depending on variable conditions such as the input torque, the input number of revolutions and the temperature of the oil, and hence is not necessarily satisfactory. Thus, it has been desired to develop a lubricating oil composition for use in a final drive which composition improves the efficiency of transmission in a differential gear under various conditions such as the input torque, the input number of revolutions and the temperature of the oil to thereby decrease the cost of fuel.

## Disclosure of the Invention

Under such circumstances, an object of the present invention is to provide a lubricating oil composition for use in a final drive for use in a car of a front-engine and rear-wheel-drive system or the like, which composition improves the efficiency of transmission in a differential gear under various conditions such as the input torque, the input number of revolutions and the temperature of the oil and hence decreases the cost of fuel, as compared with the conventional ones comprising S-containing and P-containing additives blended therein.

As a result of intensive investigations with a view to developing a lubricating oil composition for use in a final drive which composition has the foregoing favorable properties, the inventors of the present invention have found out that the above-mentioned object can be attained by a lubricating oil composition comprising a lubricant base oil, and a phosphorus-containing extreme-pressure additive, a specific organic acid and a sulfur-containing extreme-pressure additive each in a given amount. The present invention has been completed based on such a finding.

Specifically, in accordance with the present invention, there is provided a lubricating oil composition for use in a final drive characterized by comprising lubricant base oil(s); (A) 0.1 to 5 wt. %, based on the total weight of the composition, of at least one phosphorus-containing extreme-pressure additive selected from

among phosphoric esters, phosphorous esters, and alkylamine salts of phosphoric esters; (B) 0.1 to 5 wt. %, based on the total weight of the composition, of organic acid(s) represented by the general formula[1]:



(wherein  $R^1$  is an alkyl group having 1 to 18 carbon atoms, or an alkenyl group having 2 to 18 carbon atoms); and (C) 3 to 15 wt. %, based on the total weight of the composition, of at least one sulfur-containing extreme-pressure additive selected from among olefin sulfides and alkyl sulfides. The present invention will now be described in detail.

#### Brief Description of the Drawing

Fig. 1 is a schematic diagram of an apparatus as used in the LFW-1 friction test.

#### Preferable Embodiments of the Invention

Examples of the base oil to be used in the lubricating oil composition of the present invention, though not particularly restricted, include common base oils for use in the conventional lubricating oils, such as mineral and synthetic oils.

Examples of the mineral oils include 60 neutral oil, 100 neutral oil, 150 neutral oil, 300 neutral oil, and 500 neutral oil, which are prepared by refining with a solvent or through hydrogenation; and low-pour-point base oils improved in fluidity at low temperatures by removing wax from the above-enumerated base oils. These mineral oils may be used either alone or in mixture wherein at least two kinds of mineral oils are mixed together in a suitable proportion.

On the other hand, examples of the synthetic oils include  $\alpha$ -olefin oligomers, diesters, polyol esters, and polyglycol esters. These base oils may usually be used alone, but may also be used in mixture with mineral oil(s) as mentioned above. In the latter case, the mixing weight ratio of a synthetic oil to mineral oil(s) is, for example, in the range of 80:20 to 20:80.

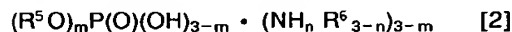
The viscosity at 100 °C of the base oil to be used in the composition of the present invention is preferably in the range of 3 to 20 cSt.

The component (A) to be used in the lubricating oil composition of the present invention is at least one phosphorus-containing extreme-pressure additive selected from among phosphoric esters, phosphorous esters, and alkylamine salts of phosphoric esters.

Examples of the above-mentioned phosphoric esters (Pa) and phosphorous esters (Pi) include a variety of phosphorus-containing compounds represented by the formula:  $\text{O}=\text{P}(\text{OR}^2)(\text{OR}^3)(\text{OR}^4)$ ,  $\text{O}=\text{P}(\text{OH})(\text{OR}^2)(\text{OR}^3)$ ,  $\text{O}=\text{P}(\text{OH})_2(\text{OR}^2)$ ,  $\text{P}(\text{OR}^2)(\text{OR}^3)(\text{OR}^4)$ ,  $\text{P}(\text{OH})(\text{OR}^2)(\text{OR}^3)$ , or  $\text{P}(\text{OH})_2(\text{OR}^2)$ . In the above-mentioned formulae,  $R^2$ ,  $R^3$  and  $R^4$ , which may be either the same or different from each other, are each a linear, branched or cyclic alkyl or alkenyl group having at least four carbon atoms, an aryl group, an alkylaryl group, or an oleylethylene oxide group.

Representative examples of these phosphoric and phosphorous esters include oleyl acid phosphate in the form of a mixture of  $(\text{C}_{18}\text{H}_{35}\text{O})\text{P}(\text{OH})_2\text{O}$  and  $(\text{C}_{18}\text{H}_{35}\text{O})_2\text{P}(\text{OH})\text{O}$ ; dioleyl hydrogen phosphite represented by the formula:  $(\text{C}_{18}\text{H}_{35}\text{O})_2\text{P}(\text{OH})$ ; and a mixture of oleylethylene oxide acid phosphates.

On the other hand, an alkylamine salt of a phosphoric ester (Pa-A) is a reaction product of the phosphoric ester with the alkylamine, examples of which include the one represented by the general formula:



In the above-mentioned general formula [2],  $R^5$  is a linear, branched or cyclic alkyl or alkenyl group having 4 to 30 of carbon atoms, preferably 20 or less of carbon atoms, an aryl group, or an alkylaryl group;  $R^6$  is a linear, branched or cyclic alkyl or alkenyl group having 4 to 30 of carbon atoms, preferably 20 or less of carbon atoms;  $m$  and  $n$  each are 1 or 2; a plurality of  $R^5$ , if any, may be either the same or different from each other; and a plurality of  $R^6$ , if any, may be either the same or different from each other.

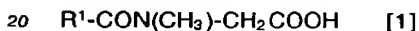
In the above-mentioned general formula [2], examples of  $R^5$  include butyl, hexyl, cyclohexyl, octyl, 2-ethylhexyl, decyl, lauryl, myristyl, palmityl, stearyl, oleyl, eicosyl, phenyl and cresyl groups; and examples of  $R^6$  include butyl, hexyl, cyclohexyl, octyl, 2-ethylhexyl, decyl, lauryl, myristyl, palmityl, stearyl, oleyl and eicosyl groups. Representative examples of the alkylamine salt of the phosphoric ester represented by the above-mentioned general formula [2] include oleylamine salt of diisooctyl acid phosphate [a reaction

product of  $(i-C_8H_{17}O)_2P(OH)O$  with  $(C_{18}H_{35})NH_2$ , and oleylamine salt of 2-ethylhexyl acid phosphate.

In the composition of the present invention, the above-mentioned phosphorus-containing extreme-pressure additives may be used either alone or in combination of two or more kinds thereof. The amount of the phosphorus-containing extreme-pressure additive(s) to be blended in the composition is in the range of 0.1 to 5 wt. %, preferably 0.5 to 3 wt. %, based on the total weight of the composition. When this amount is smaller than 0.1 wt. %, the resulting composition is never excellent in frictional properties and abrasion-proofing properties. When it exceeds 5 wt. %, the resulting improvements in such effects are not worth an increase in the amount, while a difficulty is encountered in dissolving the increased amount of the additive(s) in the base oil(s).

The phosphorus-containing extreme-pressure additive(s) as the component (A) is generally great in abrasion-proofing effect, and moreover functions as an assistant capable of promoting the effect of the sulfur-containing extreme-pressure additive. The phosphoric ester attains improvements particularly in initial (run-in) frictional properties and abrasion-proofing properties, whereas the phosphorous ester functions in such a way as to secure low frictional properties over a long period of time particularly after the run-in. On the other hand, the alkylamine salt of the phosphoric ester is excellent particularly in abrasion-proofing properties for gears.

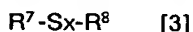
The component (B) to be used in the lubricating oil composition of the present invention is organic acid(s) in the form of derivative(s) of sarcosine represented by the general formula [1]:



In the above-mentioned formula [1],  $R^1$  is an alkyl group having 1 to 18 carbon atoms, or an alkenyl group having 2 to 18 carbon atoms, either of which may be linear, branched, or cyclic. Examples of  $R^1$  include methyl, ethyl, propyl, butyl, pentyl, hexyl, cyclohexyl, octyl, 2-ethylhexyl, decyl, lauryl, myristyl, palmityl, stearyl and oleyl groups. Among those organic acids represented by the above-mentioned general formula [1], oleyl sarcosinate is especially suitable.

In the composition of the present invention, these organic acids may be used either alone or in combination of two or more kinds thereof as the component (B), the amount of which to be blended therein is in the range of 0.1 to 5 wt. %, preferably 0.5 to 3 wt. %, based on the total weight of the composition. When this amount is smaller than 0.1 wt. %, no effect of improving the efficiency of transmission in a differential gear under various conditions such as the input torque, the input number of revolutions and the temperature of the oil can sufficiently be exhibited. When it exceeds 5 wt. %, the resulting improvement in the above-mentioned effect is not worth an increase in the amount, while a difficulty is encountered in dissolving the increased amount of the component (B) in the base oil(s).

The component (C) to be used in the lubricating oil composition of the present invention is at least one sulfur-containing extreme-pressure additive selected from among olefin sulfides and alkyl sulfides. Examples of the olefin sulfides include compounds represented by the general formula:



In the above-mentioned general formula [3],  $R^7$  is an alkenyl group having 4 to 12 carbon atoms;  $R^8$  is an alkyl or alkenyl group having 4 to 12 carbon atoms;  $R^7$  and  $R^8$  are each linear, branched, or cyclic; and  $x$  is an integer of 1 to 8. A representative example of the olefin sulfides is isobutylene sulfide ( $x=1$ ).

On the other hand, examples of the alkyl sulfides include compounds represented by the general formula:



In the above-mentioned general formula [4],  $R^9$  and  $R^{10}$ , which may be either the same or different from each other, are each an alkyl group having 4 to 12 carbon atoms, which group may be linear, branched, or cyclic; and  $y$  is an integer of 1 to 8. Representative examples of the alkyl sulfides include di-*t*-butyl disulfide and di-*t*-butyl trisulfide.

In the composition of the present invention, these sulfur-containing extreme-pressure additives may be used either alone or in combination of two or more kinds thereof as the component (C), the amount of which to be blended therein is in the range of 3 to 15 wt.%, preferably 5 to 10 wt.%, based on the total weight of the composition. The sulfur-containing extreme-pressure additive generally forms a sulfurized skin in a frictional surface, and hence functions in such a way as to improve the load resistance performance of the base oil(s). When the above-mentioned amount is smaller than 3 wt. %, the foregoing functional effect

cannot sufficiently be exhibited to make the resulting composition unsuitable as a gear oil for use in a car. When it exceeds 15 wt. %, the resulting improvement in the foregoing effect is not worth an increase in the amount, while a difficulty is encountered in dissolving the increased amount of the component (C) in the base oil(s).

5 In so far as the object of the present invention is not spoiled, the lubricating oil composition of the present invention may appropriately be admixed with a variety of common additives for use in the conventional lubricating oils, examples of which additives include a metal-cleaning agent, an ash-free cleaning dispersant, a viscosity index improver, a pour point depressant, an antioxidizing agent, a rust inhibitor, a corrosion inhibitor, and an antifoaming agent.

10 Examples of the metal-cleaning agent include calcium sulfonates, magnesium sulfonates, barium sulfonates, calcium phenate, and barium phenate. The amount of the metal-cleaning agent that may be used is usually in the range of 0.1 to 5 wt. %. Examples of the ash-free cleaning dispersant include succinimide compounds, succinamide compounds, benzylamine compounds and boron-containing derivatives thereof, and ester compounds. The amount of the ash-free cleaning agent that may be used is usually  
15 in the range of 0.5 to 7 wt. %.

Examples of the viscosity index improver include polymethacrylates, polyisobutylene, ethylene-propylene copolymers, and hydrogenated styrene-butadiene copolymers. The amount of the viscosity index improver that may be used is usually in the range of 0.5 to 35 wt. %. Examples of the antioxidizing agent include amine antioxidizing agents such as alkylated diphenylamines and phenyl-alpha-naphthylamine, and  
20 phenolic antioxidizing agents such as 2,6-di-t-butyl-4-methylphenol and 4,4'-methylenebis(2,6-di-t-butylphenol). The amount of the antioxidizing agent that may be used is usually in the range of 0.05 to 2 wt. %.

Examples of the rust inhibitor include alkenylsuccinic acids and partial esters thereof. Examples of the corrosion inhibitor include benzotriazole and benzimidazole. Examples of the antifoaming agent include dimethylpolysiloxane and polyacrylates, which may be appropriately added.

25  
Examples

The following Examples further illustrate the present invention in more detail, but should not be interpreted as limiting the scope of the invention.

30 The friction coefficient (LFW-1) and efficiency of torque transmission in a real machine of each lubricating oil composition were determined as follows.

#### (1) Friction coefficient (LFW-1)

35 A tester as shown in Fig. 1 was used together with an S-10 test ring (steel) manufactured by Falex Corporation and an H-60 block (steel) manufactured by Falex Corporation under conditions involving a sliding velocity of 1.4 m/sec, a load of 113 kgf and an oil temperature of 100 °C to carry out the LFW-1 friction test. In Fig. 1, numeral 1 refers to the S-10 test ring, numeral 2 to the H-60 block, and numeral 3 to a strain meter. The load was applied onto the H-60 block to revolve the ring, whereupon a resistance arose,  
40 which was detected with the strain meter to calculate the friction coefficient. The test oil was filled to such an extent that the ring was dipped therein by about half.

#### (2) Efficiency of torque transmission in real machine (%)

45 The test was carried out under conditions involving an input torque of 3 kgf · m, an input number of revolutions of 1,000 rpm and an oil temperature of 50 °C to determine the efficiency (%) of torque transmission in the real machine.

#### Examples 1 to 4 and Comparative Examples 1 to 6

50 Lubricating oil compositions each containing a base oil (a highly refined mineral oil having a viscosity at 100 °C of 11.0 cSt) and various kinds of components as listed in Table 1, the amounts of which are also specified in Table 1, were prepared to determine the friction coefficients (LFW-1) and efficiencies of torque transmission in the real machine of the compositions. The results are shown in Table 1.

55

Table 1

	Ex. 1	Ex. 2	Ex. 3	Ex. 4	Comp. Ex. 1	Comp. Ex. 2	Comp. Ex. 3	Comp. Ex. 4	Comp. Ex. 5	Comp. Ex. 6
Isobutylene sulfide	7.0	8.5	7.0	6.5	7.0	7.5	7.0	6.5	7.0	7.0
Oleylethylene oxide acid phosphate	1.0	2.5	2.5	2.5	-	-	-	-	2.5	-
Oleyl sarcosinate	1.0	2.5	2.5	1.5	-	-	-	-	-	2.5
Oleylamine salt of 2-ethylhexyl acid phosphate	-	-	-	-	3.0	3.0	3.0	2.5	-	-
Diolel phosphate	1.0	-	-	-	2.0	2.0	2.5	2.5	-	-
Oleyl acid phosphate	-	-	-	-	0.5	0.5	0.5	0.5	-	-
Polybutenyl succinimide	-	-	-	-	0.5	0.5	0.5	0.5	-	-
Friction coefficient (LFW-1)	0.063	0.060	0.063	0.065	0.088	0.090	0.087	0.085	0.075	0.077
Efficiency of torque transmission in real machine (%)	94.1	94.5	94.4	94.3	92.8	92.7	92.8	92.8	93.5	93.3

As is understandable from Table 1 each of the lubricating oil compositions according to the present invention was low in the friction coefficient (LFW-1) and improved in the efficiency of torque transmission in the real machine, as compared with the conventional ones comprising S-containing and P-containing additives blended therein.

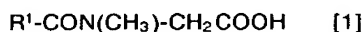


## Effects of the Invention

The lubricating oil composition of the present invention for use in a final drive, wherein base oil(s) is blended with a combination of phosphorus-containing extreme-pressure additive(s) and sulfur-containing extreme-pressure additive(s) with organic acid(s) in the form of derivative(s) of sarcosine, improves the efficiency of transmission in a differential gear under various conditions such as the input torque, the input number of revolutions and the temperature of the oil and hence decreases the cost of fuel, as compared with the conventional ones comprising S-containing and P-containing additives blended therein.

## 10 Claims

1. A lubricating oil composition for use in a final drive characterized by comprising lubricant base oil(s); (A) 0.1 to 5 wt. %, based on the total weight of said composition, of at least one phosphorus-containing extreme-pressure additive selected from among phosphoric esters, phosphorous esters, and alkylamine salts of phosphoric esters; (B) 0.1 to 5 wt. %, based on the total weight of said composition, of organic acid(s) represented by the general formula [1]:

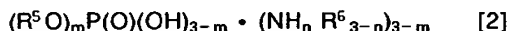


(wherein  $R^1$  is an alkyl group having 1 to 18 carbon atoms, or an alkenyl group having 2 to 18 carbon atoms); and (C) 3 to 15 wt. %, based on the total weight of said composition, of at least one sulfur-containing extreme-pressure additive selected from among olefin sulfides and alkyl sulfides.

2. The lubricating oil composition for use in a final drive characterized according to claim 1 wherein the viscosity at 100 °C of the base oil is in the range of 3 to 20 cSt.

3. The lubricating oil composition for use in a final drive according to claim 1 wherein the phosphoric ester and phosphorous ester are selected from the group of consisting of the phosphorus-containing compounds represented by the formula:  $\text{O} = \text{P}(\text{OR}^2)(\text{OR}^3)(\text{OR}^4)$ ,  $\text{O} = \text{P}(\text{OH})(\text{OR}^2)(\text{OR}^3)$ ,  $\text{O} = \text{P}(\text{OH})_2(\text{OR}^2)$ ,  $\text{P}(\text{OR}^2)(\text{OR}^3)(\text{OR}^4)$ ,  $\text{P}(\text{OH})(\text{OR}^2)(\text{OR}^3)$ , and  $\text{P}(\text{OH})_2(\text{OR}^2)$ ; wherein  $R^2$ ,  $R^3$  and  $R^4$ , which may be either the same or different from each other, are each a linear, branched or cyclic alkyl or alkenyl group having four to thirty carbon atoms, an aryl group, an alkylaryl group, or an oleylethylene oxide group.

4. The lubricating oil composition for use in a final drive according to claim 1 wherein the alkylamine salt of a phosphoric ester is the one represented by the general formula [2]:

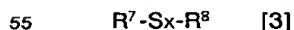


wherein  $R^5$  is a linear, branched or cyclic alkyl or alkenyl group having 4 to 30 of carbon atoms, an aryl group, or an alkylaryl group;  $R^6$  is a linear, branched or cyclic alkyl or alkenyl group having 4 to 30 of carbon atoms; m and n each are 1 or 2; a plurality of  $R^5$ , if any, may be either the same or different from each other; and a plurality of  $R^6$ , if any, may be either the same or different from each other.

5. The lubricating oil composition for use in a final drive according to claim 1 wherein  $R^1$  is selected from the group consisting of methyl, ethyl, propyl, butyl, pentyl, hexyl, cyclohexyl, octyl, 2-ethylhexyl, decyl, lauryl, myristyl, palmityl, stearyl and oleyl groups.

6. The lubricating oil composition for use in a final drive according to claim 1 wherein the organic acid is an oleyl sarcosinate.

7. The lubricating oil composition for use in a final drive according to claim 1 wherein the olefin sulfide is the compound represented by the general formula [3]:



Wherein  $R^7$  is an alkenyl group having 4 to 12 carbon atoms;  $R^8$  is an alkyl or alkenyl group having 4 to 12 carbon atoms;  $R^7$  and  $R^8$  are each linear, branched, or cyclic; and x is an integer of 1 to 8.

8. The lubricating oil composition for use in a final drive according to claim 1 wherein the olefin sulfide is an isobutylene sulfide.

5 9. The lubricating oil composition for use in a final drive according to claim 1 wherein the alkyl sulfide is the compound represented by the general formula [4]:



10 wherein  $R^9$  and  $R^{10}$ , which may be either the same or different from each other, are each an alkyl group having 4 to 12 carbon atoms, which group may be linear, branched, or cyclic; and y is an integer of 1 to 8.

15 10. The lubricating oil composition for use in a final drive according to claim 1 wherein the olefin sulfide is a di-t-butyl disulfide or a di-t-butyl trisulfide.

11. The lubricating oil composition for use in a final drive according to claim 1 which comprises a variety of the conventional additives for use in the lubricating oils.

20 12. The lubricating oil composition for use in a final drive according - to claim 11 wherein the additives are selected from the group consisting of a metal-cleaning agent, an ash-free cleaning dispersant, a viscosity index improver, a pour point depressant, an antioxidizing agent, a rust inhibitor, a corrosion inhibitor, and an antifoaming agent.

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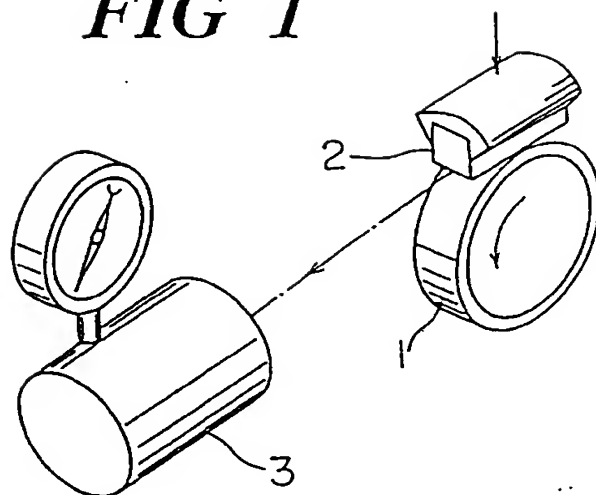
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**FIG 1**



## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/JP93/01918

## A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl<sup>5</sup> C10M141/10// (137:02, 133:16, 135:04) C10N40:04

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int. Cl<sup>5</sup> C10M141/10, 137:02, 133:16, 135:04, C10N40:04

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y A	JP, A, 2-298590 (Ethyl Petroleum Additives, Ltd.), December 10, 1990 (10. 12. 90), Claim, line 13, upper right column, page 3 to line 3, upper right column, page 5 & EP, A2, 391653	1-12
A	JP, A, 2-182787 (Tonen Corp.), July 17, 1990 (17. 07. 90), Claim, line 6, lower right column, page 3 to line 19, upper right column, page 4, (Family: none)	1-12
A	JP, A, 63-61091 (Showa Shell Sekiyu K.K.), March 17, 1988 (17. 03. 88), Claim, line 10, lower left column, page 2 to line 20, upper right column, page 3, (Family: none)	1-6, 9-12
A	JP, A, 61-241396 (Ajinomoto Co., Inc.), October 27, 1986 (27. 10. 86),	1

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

## \* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

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"Z" document member of the same patent family

Date of the actual completion of the international search

March 1, 1994 (01. 03. 94)

Date of mailing of the international search report

March 22, 1994 (22. 03. 94)

Name and mailing address of the ISA/

Japanese Patent Office

Facsimile No.

Authorized officer

Telephone No.

**INTERNATIONAL SEARCH REPORT**

International application No.

PCT/JP93/01918

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	Claim, (Family: none)	

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